#### TECHNICAL STANDARD OPERATING PROCEDURE

Date: August 26	5, 1999		SOP No.	MK-VBI70-05
Title: Sample I	Preparation			
APPROVALS	:			
Author:	Morrison Knud	sen Corporation	Date:	August 26, 1999
*	ovides procedures a laboratory analysi	and instructions for the prepas.	ration of soil sa	mples for on-site
Received by QA	<u>Unit</u>			
REVIEWS:				
TEAM MEMBI	<u>ER</u>	SIGNATURE/TITLE		DATE
EPA Region 8	Bo	wh Josh /RI	endinator	8/27/99
Morrison Knudse	en Corp.	e Met 10A COO	<u>Adirator</u>	9/26/99
DEV	DATE	DEVISION	I DESCRIPTION	

REV.	DATE	REVISION DESCRIPTION
1	8/4/99	Grinding of bulk soil prior to XRF analysis added
2	8/26/99	Sieving sample to be performed after drying instead of prior to drying



#### 1.0 PURPOSE

The purpose of this procedure is to provide instructions to Morrison Knudsen personnel assigned to the VB/I-70 Project and their subcontractors for the preparation of soil samples.

#### 2.0 SCOPE

This procedure covers activities associated with preparation of soil samples for subsequent analysis by X-ray fluorescence spectrometry, inductively coupled plasma spectroscopy, and/or bioavailability tests.

#### 3.0 REFERENCES

Method 6200 Field Portable X-Ray Fluorescence Spectrometry For The Determination Of Elemental Concentrations In Soil And Sediment

Spectrace QuanX Laboratory X-Ray Fluorescence Analyzer Standard Operating Procedure

Standard Operating Procedure for Chain of Custody and Sample Handling, SOP No. MK-VBI70-02

Standard Operating Procedure for Equipment Decontamination, SOP No. MK-VBI70-07

Standard Operating Procedure for Investigation Derived Waste Management, SOP No. MK-VBI70-04

#### 4.0 **DEFINITIONS**

None



#### 5.0 RESPONSIBILITIES

The **Sample Preparation Technician** will be responsible for overseeing sample receipt and chain of custody both before and after the preparation process, and implementation of the sample preparation process.

The **Field Supervisor** will be responsible for quality and production of field laboratory operations.

The Site Health and Safety Officer will be responsible for verifying implementation of this procedure using safe laboratory practices.

The Field Quality Assurance Coordinator will be responsible for overseeing proper implementation of the quality control procedures, including tracking of blind standard samples, tracking of confirmation samples for off-site laboratory analysis, and specification of sample labels to be used for blind split samples.

The **Site Manager** will be responsible for ensuring that personnel are properly trained to this procedure.

#### 6.0 REQUIREMENTS

#### 6.1 General

- 6.1.1 Sample preparation activities shall be performed only in areas designated for each activity.
- 6.1.2 Eating and smoking are prohibited in all areas of the sample preparation area.
- 6.1.3 Samples generally will be prepared in batches consisting of twenty field samples.

SOP No. MK-VBI70-05

6.1.4 All non-dedicated equipment used during sample preparation must be decontaminated prior to use as described in the Decontamination SOP (MK-VBI70-07).

#### 6.2 Equipment

Sample drying trays

Permanent Marking pen

General purpose laboratory oven

#10 mesh stainless steel sieve

#60 mesh stainless steel sieve

Sample bags

XRF cups

Mylar

Spatulas

Stainless steel spoon

Analytical balance accurate to 0.1 g, range of 0.1 g to 1000 g

Mortar and pestle, 140 mL or greater (or mill equipped with Burundum cylinders)

#### 6.3 Soil Mixing

- 6.3.1 Select samples to be prepared. Prior to opening the sample bag, knead the contents to break up soil clumps and mix approximately two minutes or until the soil appears to be well homogenized. If the kneading process produces cohesive clumps, that observation will be noted in the Preparation Log.
- 6.3.2 Mix by turning the bag end over end slowly a minimum of ten times, then using a large stainless steel spoon, stir the contents of the sample bag thoroughly.

#### 6.4 Bulk Soil Drying

6.4.1 Set the oven temperature to 103-105 C (not to exceed 115 C). Record the oven temperature at least once daily in the Sample Preparation logbook.

- 6.4.2 Pour approximately 6 ounces of the sample into a pre-labeled (3-XXXXX-R) drying pan and pour the remainder of the soil into a pre-labeled bag for archiving (3-XXXXX-RA) under chain of custody documentation. Spread the sample on the drying tray in an even layer to promote even drying.
- 6.4.3 Check the oven temperature to verify proper temperature has been reached. Place the drying trays containing the samples into the oven(s). Leave the samples in the oven until completely dry as defined by a stable sample weight. Establish the drying time initially by recording weights for samples with varying soil moisture: 1) before drying, 2) at estimated completion, and 3) following an additional 15 minute drying time to confirm stable weight. Verify sample dryness for all samples by squeezing a portion of the sample between a gloved thumb and forefinger. Sample dryness is indicated by a lack of cohesiveness in the soil. Document the sample drying time for each sample on the Sample Preparation Log.
- 6.4.4 When samples are dry, remove from the oven and place in the ventilation area. **Before** placing samples at the ventilation area, verify that the blower is turned on.

#### 6.5 Bulk Sieving

- Pour the sample from the drying pan onto a #10 sieve attached to a catch pan. Shake the sieve to pass the sample through the sieve into the catch pan. Dispose of any sample that did not pass through the sieve into the waste soil receptacle.
- 6.5.2 Place the sieved sample into a pre-labeled (3-XXXXX-B) sample bag. Completely seal the bag, then mix by turning the bag end over end slowly a minimum of ten times.
- 6.5.3 Document the date sieving was performed for each sample in the Sample Preparation Log Sheet.

#### 6.6 Bulk Soil Grinding and Cupping

- 6.6.1 Using a spatula, stir the contents of the sample bag thoroughly, then transfer approximately 10 grams to the mortar.
- 6.6.2 Grind the soil using the pestle for ten minutes or until all material is evenly ground to a powder. If larger grained vegetation or soil materials remain, sieve the sample through a #60 sieve.
- 6.6.3 Using a spatula, fill the pre-labeled (3-XXXXX-B) XRF cup with soil from the mortar, filling cup ½ to ¾ full. Secure a piece of Mylar film over the top of the cup to seal.
- 6.6.4 Prepare XRF quality control samples as described in Section 6.8.2. Dispose of any unused ground soil into the waste soil receptacle.

#### 6.7 Drying and Sieving Fine Fraction Soil

- 6.7.1 Selected archived bulk soil samples will be dried at a low temperature and sieved to isolate the naturally occurring fine fraction using a #60 mesh sieve.
- 6.7.2 Set the oven temperature to 45-48 C (not to exceed 50 C).
- 6.7.3 Pour approximately 8 ounces of soil onto a pre-labeled (3-XXXXX-RA) drying tray and spread in an even layer to promote even drying. Return the remaining soil to the archive.
- 6.7.4 Check the oven temperature to verify proper temperature has been reached. Place the drying trays containing the samples into the oven(s). Leave the samples in the oven until completely dry as defined by a stable sample weight. Establish the drying time initially by recording weights for samples with varying soil moisture: 1) before drying, 2) at estimated completion, and 3) following an additional 15 minute drying time to confirm stable weight. Confirm sample dryness for all samples by squeezing a portion of the sample between a gloved thumb and forefinger. Sample dryness is indicated by a lack of

cohesiveness in the soil. Document the sample drying time for each sample on the Sample Preparation Log.

- 6.7.5 When samples are dry, remove from the oven and place in the ventilation area. **Before** placing samples at the ventilation area, verify that the blower is turned on.
- 6.7.6 Pour the dried sample onto a #60 sieve attached to a catch pan. Shake the sieve to pass the sample through the sieve into the catch pan. Dispose of any sample that did not pass through the sieve into the waste soil receptacle. Place the sample in the catch pan into a pre-labeled (3-XXXXX-F) sample bag. Completely seal the bag then mix by turning the bag end over end slowly a minimum of ten times. Using a spatula, stir the soil thoroughly and then fill the pre-labeled (3-XXXXX-F) XRF cup with soil from the sample bag, filling cup ½ to ¾ full. Secure a piece of Mylar film over the top of the cup to seal.

#### 6.8 Quality Control Sample Preparation Procedure

- 6.8.1 Sample preparation will be performed in an area separate from the XRF operations. The sample preparation technician who prepares sample batches containing blind quality control samples may not perform analysis on those samples. The XRF analyst will not observe the sample preparation and will not view the preparation logs in order to maintain sample anonymity to the analyst.
- 6.8.2 Prepare each of following quality control samples at rate of one per twenty field samples by filling two XRF cups with soil (following sample drying, sieving, mixing and grinding procedures):
  - One laboratory duplicate, labeled 3-XXXXX-B(or -F)-DUP
  - One blind field split, labeled with a unique sample ID from the labels designated for QC samples.
- 6.8.3 Prepare confirmation samples at a rate specified by the EPA Remedial Project Manager (initially one per three field samples) by transferring approximately 4 ounces from the

prepared field sample bag (following drying, bulk sieving, fine sieving where applicable, and mixing; grinding of confirmation samples is <u>not</u> necessary) into a second bag and labeling the confirmation sample with the identical sample identification (i.e., 3-XXXXX-B or 3-XXXXX-F). The confirmation sample will be submitted under chain of custody to an off-site laboratory for analysis by Method 6010B (ICP) as described in the Chain of Custody and Sample Handling SOP (MK-VBI70-02).

- 6.8.4 Document the laboratory duplicate in the "Notes" column of the Field Sample
  Preparation Log. Document the blind field split Sample ID and original Sample ID on the
  QC Data Sheet for Blind Soil Field Splits.
- 6.8.5 Prepare blind standards as directed by the Field Quality Assurance Coordinator by filling a pre-labeled XRF cup with soil from the blind standard sample provided. If the standard is not received pre-dried, sieved and ground, the sample will be prepared in accordance with mixing, drying, sieving, and grinding procedures detailed above in Section 6.3, 6.4, 6.5, and 6.6. Label the cup with a unique sample ID from the list of sample labels designated for QC samples. Document the blind standard when prepared on the Performance Evaluation Standard (Blind Standard) QC Data Sheet.
- 6.8.6 Place 18 XRF cups for a single sample run into a staging container for transfer to the XRF Analyst (the analyst will complete the run with addition of a standard reference material and instrument blank).

#### 6.9 Investigation Derived Waste Management

- 6.9.1 Remove the sample receptacle from under the ventilation hood and dispose of its contents into the waste soil drum when full and at the end of each day.
- 6.9.2 Place all non-dedicated sample drying trays, sieves, catch pans, spatulas, and spoons used during sample preparation in the receptacles for equipment decontamination.

#### 7.0 ATTACHMENTS

Field Sample Preparation Logbook Sheet

QC Data Sheet, Blind Soil Field Splits

QC Data Sheet, Blind Performance Evaluation Samples

#### **ATTACHMENTS**

FIELD SAMPLE PREPARATION LOGBOOK SHEET

QC DATA SHEET, BLIND SOIL FIELD SPLITS

QC DATA SHEET, BLIND PERFORMANCE EVALUATION SAMPLES

### **VBI70 Field Sample Preparation Logbook Sheet**



ID Batci	Prep Batch	Confirmation Sample	Drying						Sieving		Notes	
	Number	Number	Date/Time Drying Begun	Initial Weight	Date/Time Drying Estimated to	Weight		Final Weight	Date Sieved	Particle Size Fraction		
				S.	be Complete					Bulk (<2mm)	Fine (<250 μm)	
				14.5								
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### **VBI70 Field Sample Preparation Logbook Sheet**



Sample F	Prep Batch Number	Drying		Sieving			Confir- mation	Notes
		Date/Time Drying Begun	Drying Drying			cle Size	Sample (Y/N)	
					Bulk (<2mm)	Fine (<250μm)		
						-		
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					-			

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# **VBI70 QC Data Sheet Blind Field Split Samples**



Date	Sample ID	Sample Class	Original Sample	Prepared By	Notes
		BD			
	:	BD			
		BD			

# VBI70 QC Data Sheet Blind Standard



Date S	Sample ID Sample Class	Sample	Lot Number	Analy	Analysis Type				
		Class		XRF	Confirmation	Ву			
		PE							
		PE							
		PE							
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